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INVESTIGATION OF THE DISTRIBUTION OF LEAD IN THREE DIFFERENT COMBINATIONS OF BRASS FEEDSTOCK

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Abstract

The main objective of this paper was to assess three leaded brass samples (pending application with Copper Development Association) using optical microscopy and mass spectrometry to compare the distribution of lead. Based on the mass spectrometry data, a great deal of variation was not found within each of the samples based on five different sample locations. Optical microscopy, scanning electron

microscopy and energy-dispersive X-ray spectroscopy confirmed that the lead was homogeneously distributed in brass.

Keywords: continuous casting, lead, brass, mass spectrometry, metallography

Introduction

Brass is extensively used in numerous market applications such as screws, valves, bearings, fittings and specialty fasteners due to its beneficial corrosion resistance, thermal and electrical conductivity, formability and good mechanical properties.¹ Some alloying elements enhance the special characteristics of brass. Lead is one of the most important elements, which can be added to any brass to increase machinability with respect to low melting point of lead and very low solubility of lead in brass. However, other elements such as bismuth (Bi), tin (Sn) and arsenic (As) are used to improve some characterisation of brass.^{2,3} Leaded brass rods can be produced by continuous extrusion forming technology and continuous casting. The main disadvantage of producing a brass alloy rod by continuous extrusion forming technology is the quality of the brass alloy rod.

The use of continuous casting gives a range of advantages in comparison with continuous extrusion forming such as low energy consumption, high productivity, length size of final product and cost.^{4,5} Leaded brass bars with the same

composition but with different combinations have been characterised in this work. Chemical composition and microstructure have been studied in order to clarify the distribution of lead (Pb).

Brass Phase Diagram

Figure 1 shows the phase diagram of brass. CuZn alloy system contains intermediate phases. Brass alloys having various Zn content are categorised into different types of brass for example:

1. Alpha brasses [Zinc (%) <35], which contain only one phase, with face-centred cubic (FCC) crystal structure.
2. Alpha-beta brasses [Zinc (%) 35–45] which contains both α and β' phase; β' phase is body-centred cubic (BCC) and α phase is FCC.
3. Beta brasses [Zinc (%) 45–50], which contain only one phase, with BCC crystal structure.

Leaded brass is an alpha-beta brass with an addition of lead with excellent machinability.

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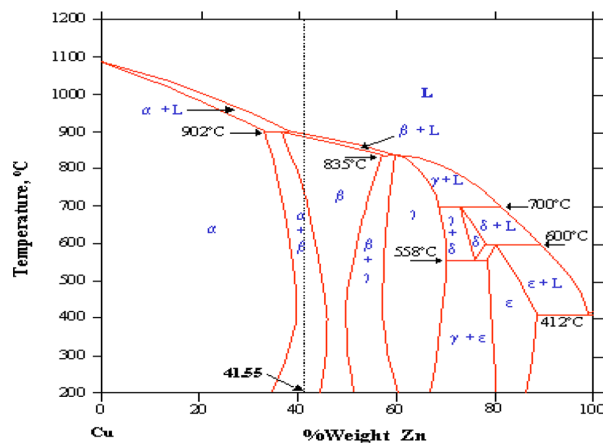


Figure 1. Phase diagram of brass.

Table 1. Leaded Brass Samples Tested in this Research

Sample name	Combination
Sample 1	Cu granules and Zn pieces melted in an induction furnace together with elemental additions and poured into a Rautomead caster
Sample 2	Brass scrap and Zn pieces together with the balance of elemental additions melted and cast in a Rautomead caster
Sample 3	Cu scrap and Zn pieces together with the elemental additions melted and cast in Rautomead caster

Experimental Procedure

Leaded Brass Samples

The representative leaded brass samples analysed in this work and their corresponding combination and information is listed in Tables 1, 2 and 3. Leaded brass samples with 0.1–0.2 % Pb contents according to Table 4 were used in this investigation. Nearest Alloy Designation is C28XXX (pending application with Copper Development Association—CDA). Distribution of Pb was investigated by metallography and mass spectrometry analysis.

In this work the charge was first weighed by the operator and then was melted in a graphite crucible using graphite heating element furnace technology (Rautomead horizontal continuous casting machine) as shown in Figures 2a, b and 3.

Metallography

Samples for metallographic examination were prepared by conventional techniques. Metallographic sections were cut with a clean sharp hacksaw and then ground using alumina grinding paper, first using coarse abrasive paper (grit no. 220) and subsequently wet and dry fine paper (grit no. 1200) by

Table 2. Leaded Brass Sample Data

Sample name	Rod dia (mm)	Continuous casting types
Sample 1	28	Horizontal
Sample 2	28	Horizontal
Sample 3	28	Horizontal

5–10 lbs force and water as a lubricant. Base/head speed of grinding was 100/100 rpm. The samples were then polished using diamond paste beginning with 1 micron and then subsequently using ¼ micron. Base/head speed of polishing was 100/100 rpm and the force was 5–10 lbs. After polishing, the samples were cleaned by acetone in an ultrasonic cleaner and dried with nitrogen gas. According to the ASTM E407-07 (Standard Practice for Micro-Etching Metals and Alloys), the polished samples were etched in a solution of 70 % concentrated nitric acid (HNO₃) and water. Samples structure was investigated using a Keyence VHX 3D digital microscope.

Mass Spectrometry Analysis

Mass spectrometry (MS), wet chemistry including gravimetric and titrimetric techniques, spark optical emission spectroscopy (Spark-OES), inductively coupled plasma-optical emission spectroscopy (ICP-OES), X-ray fluorescence (XRF) and X-ray diffraction (XRD) are common analytical chemistry techniques to identify the amount and type of chemicals present in a sample. The mass spectrometer has a few advantages over the other analytical methods such as small sample size, accuracy, fast analysis and less demanding safety issues as compared to the X-ray techniques.^{6–9} In this research mass spectrometry was used (model: AMETEK) as an analytical technique to identify the amount of chemicals present in samples. The samples were prepared using a milling machine. Figure 4 shows photographic images of samples before, during and after mass spectrometry analysis.

SEM/EDX

A scanning electron microscope was employed to produce high resolution images. Energy-dispersive X-ray spectroscopy analysis is a well-known X-ray technique used to identify the elemental composition of materials. In this report a JEOL JSM7400F field emission scanning electron microscope was used to identify the ‘dark spots’ assumed to be Pb particles, which are the feature of interest.

Results and Discussion

Results and Discussion from Optical Microscope

The grain structure of the leaded brasses is similar to the unleaded brasses.¹⁰ The microstructures of the leaded

Table 3. Leaded Brass Sample Charge Weight


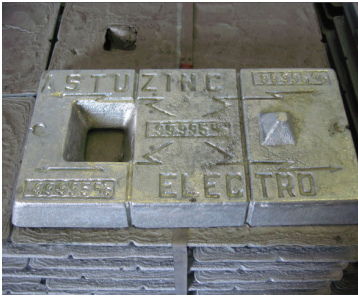

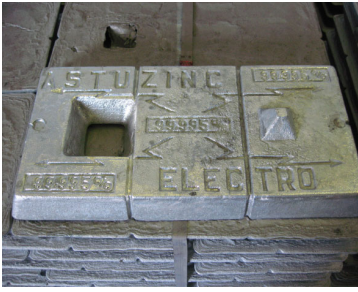


Sample name	Charge weight	Picture of the charge	Picture of the Zn piece
Sample 1	Granules into approximately 5 kg briquettes Zn in 25 kg slabs		
Sample 2	Various brass scrap from approximately 500 g–5 kg Zn in 25 kg slabs		
Sample 3	Copper scrap various from a few grams to 50 kg Zn in 25 kg slabs		

Table 4. Leaded Brass Sample Target Range

Element	Target range	UNS number
Bi	0.6–0.7	Nearest Alloy Designation is C28XXX (pending application with Copper Development Association—CDA)
Sn	0.3	
Pb	0.1–0.2	
As	0.09–0.13	
Zn	36.6–37.5	

brasses contain lead particles mainly in the grain boundaries or inter-dendritic regions. Lead is practically insoluble in solid copper and appears as a dark particle in the structure.¹¹ In order to identify lead, the samples were examined under a digital optical microscope—magnification 1000×. Figure 5 shows the typical equiaxial grain

morphology structure of brass samples and highly insoluble Pb as a dark particle. The Pb content was too low to draw any important conclusion about the homogeneity of Pb in the samples. However, it loosely appeared to be fairly well distributed.

Results and Discussion from Mass Spectrometry Analysis

The work was continued using the AMETEK spectrometer to examine different areas on each specimen. Areas 1 through 4 are around the circumference of the sample, and area 5 is in the middle. Results show that there is no significant difference in the distribution of any one of the elements based on the mass spectrometry result. Table 5 and Figure 6 present the findings.



(a) Weighing scraps by operator



(b) Scraps

Figure 2. Weighing by operator.

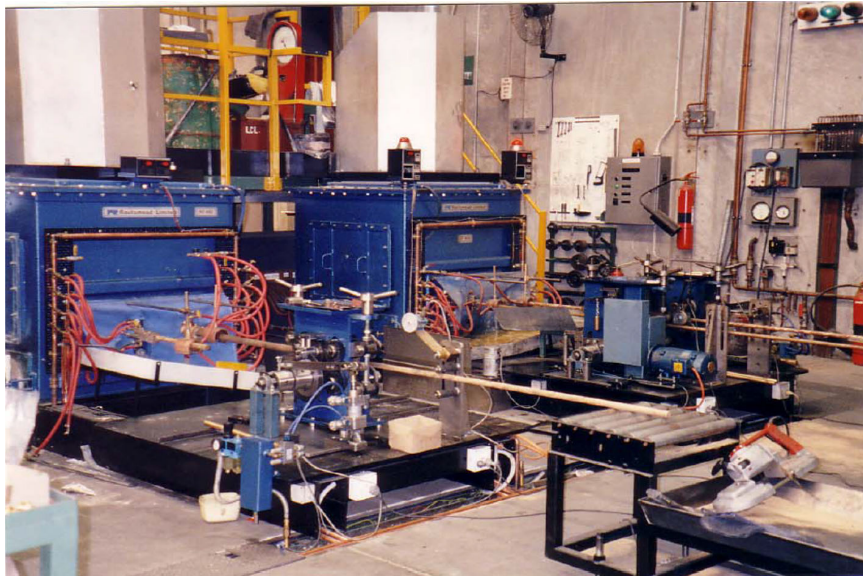
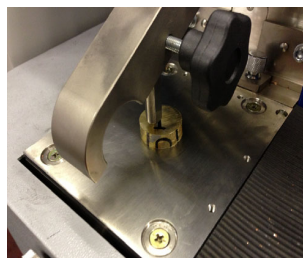


Figure 3. Horizontal continuous casting.



(a) Samples before mass spectrometry analysis



(b) Samples during mass spectrometry analysis



(c) Samples after mass spectrometry analysis

Figure 4. Spectrometry analysis. (a) Samples before mass spectrometry analysis. (b) Samples during mass spectrometry analysis. (c) Samples after mass spectrometry analysis.

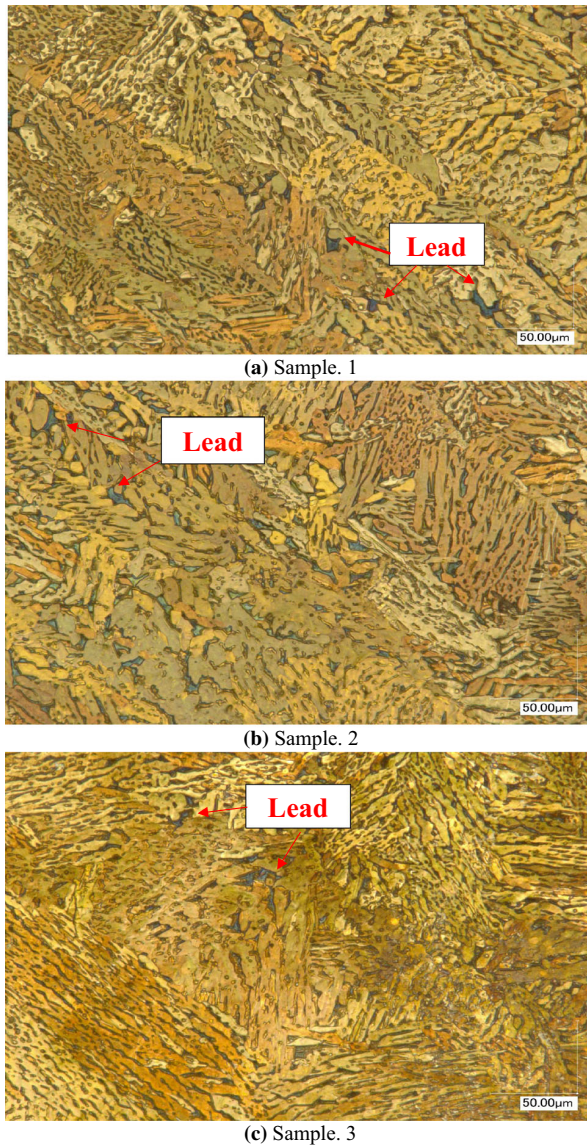


Figure 5. Cross section of the microstructure of leaded brass sample.

Analysis of variance (ANOVA) was carried out on the samples to observe any difference between the groups on some variable using ANOVA. ANOVA is a statistical procedure to analyse the difference between two or more means and is used to analyse general alternately specific difference between means.¹² As we see in the above chart, we have three groups of samples, labelled 1, 2, 3. There is a target range for samples: the aim is that the sample should be located within this range, and then the statistical method of ANOVA is applied over these samples. From this analysis, the results fall perfectly in the target range, i.e. the ANOVA analysis of variance failed to reject the null hypothesis with a confidence level of $\alpha = 0.05$. This means that there is no proven significant difference in any of the

Table 5. Leaded Brass Mass Spectrometry Results

Location	Zn	Pb	Sn	As	Bi
<i>Sample no: 1</i>					
1	35.956	0.187	0.338	0.142	0.690
2	36.036	0.188	0.333	0.140	0.685
3	36.530	0.187	0.331	0.139	0.681
4	36.456	0.187	0.328	0.138	0.681
5	36.476	0.183	0.330	0.136	0.678
<i>Sample no: 2</i>					
1	37.073	0.200	0.314	0.115	0.714
2	36.836	0.199	0.314	0.115	0.713
3	36.866	0.198	0.314	0.114	0.714
4	37.163	0.199	0.313	0.115	0.715
5	37.120	0.196	0.312	0.113	0.704
<i>Sample no: 3</i>					
1	36.316	0.140	0.279	0.101	0.669
2	36.580	0.124	0.277	0.100	0.677
3	36.690	0.123	0.276	0.099	0.675
4	36.643	0.123	0.277	0.099	0.673
5	36.316	0.124	0.278	0.100	0.676

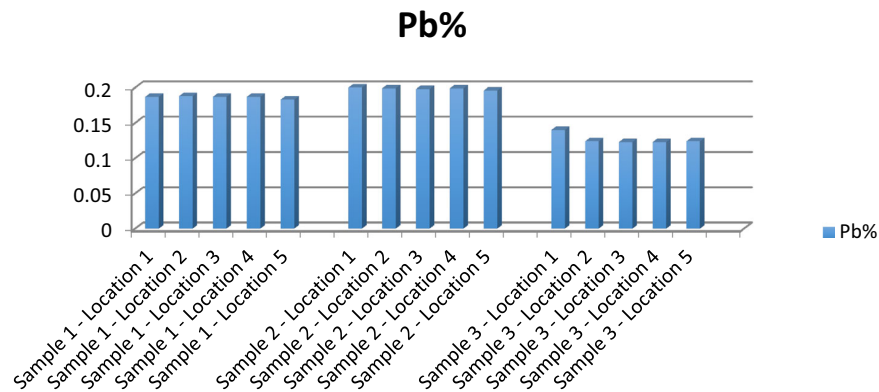


Figure 6. Leaded brass mass spectrometry results.

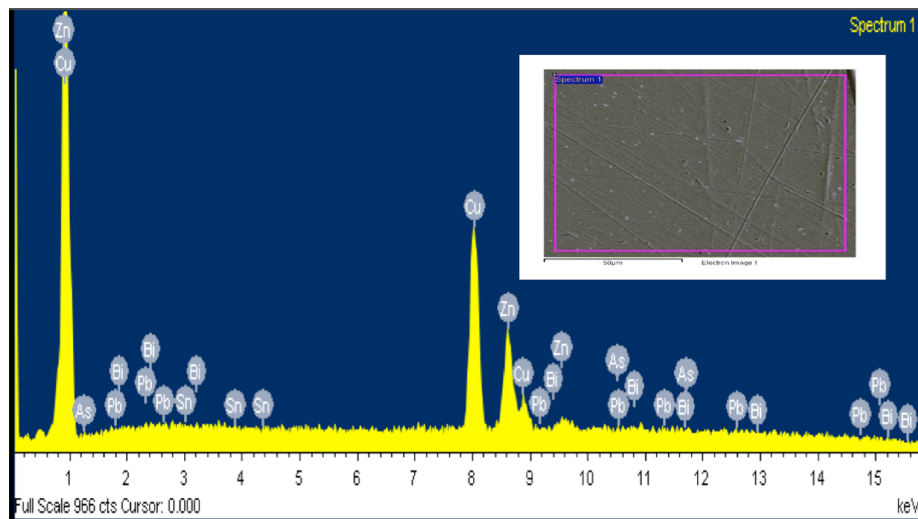


Figure 7. SEM and EDX of overall feature.

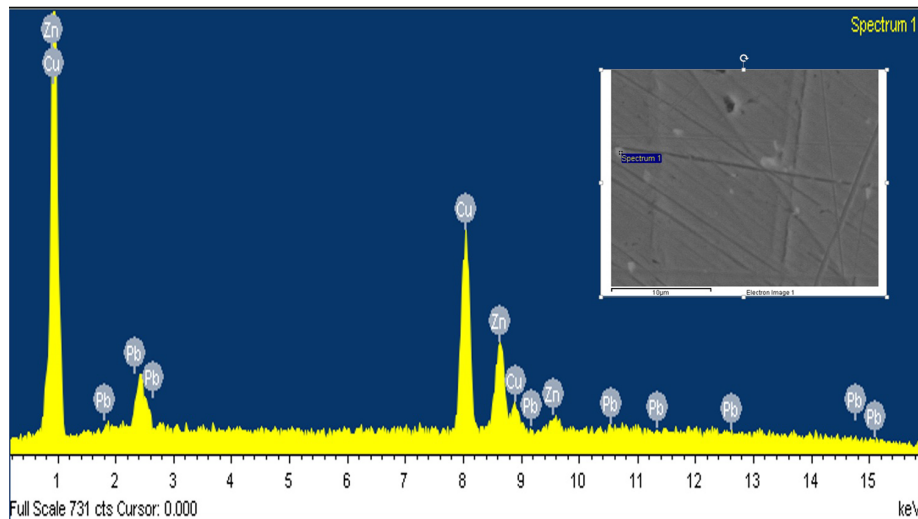


Figure 8. SEM and EDX of bright particle.

samples tested on the spectrometer based on their respective areas of testing.

Results and Discussion from SEM Image Observation and EDX Analysis

Figures 7 and 8 show the structure of brass samples. The peaks corresponding to the elements, which were generated by the EDX analysis, show the overall texture of the sample to be Cu and Zn. In addition, the bright particles have been determined using selected point area EDX analysis and Pb was identified. The SEM/EDX analysis on the samples demonstrated that the distribution of elements inside each individual sample is homogeneous, which confirms the result from the optical microscope and mass spectrometry analysis.

Conclusions and Future Work

The obtained results regarding the investigation of the distribution of lead in three different combinations of brass feedstock by mass spectrometer, digital optical microscope and SEM/EDX can be summarised in the following points:

1. Following on from the mass spectrometry results, the distribution of elements inside each individual sample is homogeneous.
2. Homogenised distribution of lead in three different combinations of brass feedstock by mass spectrometer has been confirmed by ANOVA analysis.
3. Regarding the optical microscopy and SEM and EDX analysis done on the samples, the

distribution of lead inside each individual sample is homogeneous.

4. Based on mass spectrometry we could not find a great deal of variation within each samples based on sampling five different locations.
5. As a future work, this work can link the observation of the surface area oxide from granules versus larger chunks.
6. As for future work, this research can be extended to investigate lead using X-ray fluorescence (XRF) machine, which is categorised as non-destructive analysis.

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